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Agricultural Research Institute, Pusa

STANDARD METHODS OF ANALYSIS OF FERTILIZERS

Edited by

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PREFACE

Reports on the composition of agricultural products at times show relatively wide divergences, even when the analyses are carried out by specialists, unless the same standard methods are adopted. In view of this fact, the First Conference of Agricultural Chemists and Bacteriologists, held at Pusa in 1919, adopted a resolution recognizing the necessity for the standardization of methods of analysis. A Standing Committee was appointed to investigate and report on methods applicable to specific materials. Investigation of each of the fertilizer constituents was distributed to sets of workers chosen from among the members of the Committee, of which Dr. W. H. Harrison, Imperial Agricultural Chemist, was appointed Secretary. It was decided that reports on critical studies of existing methods and of the new methods as they appear were to be transmitted to the Secretary for circulation.

The reports were considered at the Second Conference of Chemists and Bacteriologists which met at Pusa in 1921, when certain methods were accepted as official, while certain others were temporarily adopted as provisional. A Committee was formed to prepare and circulate details of the proposed standard methods for analysis of fertilizers. It was hoped that before the next meeting of the Conference, which was to take place in 1923, it would be possible to thoroughly test these methods in the different Agricultural Chemical Laboratories, and that it would thus be possible to have all the standard methods in shape for final acceptance at the Conference. As unfortunately no Conference of Chemists and Bacteriologists has been held since 1921, the Imperial Agricultural Chemist undertook to circulate the final report to agricultural chemists and other scientists interested in the subject throughout India. All the members of the postponed Conference of 1923 were requested to furnish the Imperial Agricultural Chemist with their opinion or any suggestions they wished to offer. Cordial thanks are due to the members of the Committee most of whom have kindly responded to the request. The consensus of opinion being in favour of publication of these methods, this is now being done, after considering the individual points raised.

In conclusion, it may be pointed out that the methods are largely based on the Official and Tentative Methods of Analysis published in 1920 by the Association of Official Agricultural Chemists of the United States of America. The value of this monumental work of the Association has been universally acknowledged. As, however, the conditions

in India are somewhat different from those prevailing in America, it has been necessary to modify the procedure in certain details. Needless to say, ease of manipulation together with quickness of operation have been taken into consideration along with accuracy of the results obtained.

J. SEN.

CONTENTS

		Page
	SAMPLING	
1	COLLECTION OF GROSS SAMPLE	1
2	SAMPLING FOR ANALYSIS	*ib.*
3	MECHANICAL ANALYSIS OF BONE MEAL, ROCK PHOSPHATE AND BASIC SLAG	2
4	MOISTURE	*ib.*
	TOTAL PHOSPHORIC ACID	*ib.*
	Gravimetric Method	*ib.*
5	REAGENTS	*ib.*
6	PREPARATION OF SOLUTION	3
7	DETERMINATION	4
8	*Volumetric Method*	*ib.*
9	REAGENTS	*ib.*
10	PREPARATION OF SOLUTION	5
11	DETERMINATION	*ib.*
12	WATER-SOLUBLE PHOSPHORIC ACID	*ib.*
	CITRATE-INSOLUBLE PHOSPHORIC ACID	6
13	PREPARATION OF AMMONIUM CITRATE SOLUTION	*ib.*
14	DETERMINATION	*ib.*
15	CITRATE-SOLUBLE PHOSPHORIC ACID	*ib.*
	ORGANIC AND AMMONIACAL NITROGEN ONLY	7
	Kjeldahl Method	*ib.*
16	REAGENTS	*ib.*
17	DETERMINATION	8
	TOTAL NITROGEN	*ib.*
	Kjeldahl Method modified to include the Nitrogen of Nitrates	*ib.*
18	REAGENTS	*ib.*
19	DETERMINATION	9
20	AMMONIACAL NITROGEN	*ib.*
21	NITRIC AND AMMONIACAL NITROGEN	*ib.*
	POTASH	10
	Platinum Method	*ib.*
22	REAGENTS	*ib.*
23	PREPARATION OF SOLUTION	*ib.*
24	DETERMINATION	*ib.*
	Perchlorate Method	11
25	REAGENTS	*ib.*
26	PREPARATION OF SOLUTION	13
27	DETERMINATION	*ib.*

Standard Methods of Analysis of Fertilizers.

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SAMPLING.

1 COLLECTION OF GROSS SAMPLE.

From a heap this is best done by the following procedure. Dig well into the heap from several directions and take spade-fulls at regular distances apart. When sampling has to be done from sacks containing material in a firm state, take complete sections from top to bottom by means of the sampling tool (a cylinder of iron 1 inch in internal diameter, sharpened at one end and having a slit from the bottom to near the top, where a handle is attached). In the case of manures in a powdered condition, lay the sacks on their sides and take out samples with the help of the above-mentioned tool. When sampling oil cake, see that the sample is uniform in appearance. If it is not uniform, collect the sample only after carefully grading it. For this purpose select typical cakes, break them in halves and cut off a section 2 inches wide from the middle of each. Break up the cake sections for further sampling.

2 SAMPLING FOR ANALYSIS.

(*a*) If the gross sample is moist and cannot be air-dried without danger of loss occurring to some of the ingredients to be determined, rapidly mix and quarter the sample. When necessary, take such special steps as are required to avoid changes of composition. For example, if loss of ammonia is feared, add sulphuric acid to part of the final sample. In such cases always enter in the report a full description of the state of the manure when sampled and the procedure adopted to avoid loss.

(*b*) If the gross sample is not air-dry and if air-drying will not cause loss of the ingredients to be determined, weigh the gross sample. Air-dry it and reweigh.

Reduce the air-dry gross sample by quartering to an amount sufficient for analytical purposes.* If a mechanical analysis has to be carried out,

* If the analysis is required in evidence before a court of law, the sampling as prescribed must be carried out in the presence of a witness and at this stage three bottles of sample filled, closed and sealed. The bottles are to be distributed as follows:—one to the seller, one to the analyst, one to the Director of Agriculture. The analyst, on receiving such a sample, will, before opening it, examine the label and seal and enter all details regarding these in his sample register.

as in the case of bone meal and rock phosphate, a part should be set aside for this.

Transfer to a sieve having circular openings 1 mm. in diameter. Sift, breaking up the lumps with a rubber pestle. Grind in a mortar the part remaining on the sieve until the particles pass through. Mix well and preserve in a stoppered bottle.

3 MECHANICAL ANALYSIS OF BONE MEAL, ROCK PHOSPHATE AND BASIC SLAG.

Transfer 100 grm. of the material to a sieve having circular holes 0·5 mm. in diameter. Sift, breaking up the lumps with a soft rubber pestle. Weigh the coarse portion remaining on the sieve. Determine the fine portion by difference.

4 MOISTURE.

(*a*) If the sample cannot be air-dried, special steps should be taken to determine the moisture in the gross sample and in the portions used for analysis.

(*b*) In the case of air-dry samples, heat 2-5 grm. of the sample in a water oven at the temperature of boiling water for five hours, or more, till the weight is constant. For the determination of moisture in potash salts, sodium nitrate and ammonium sulphate, heat at about 130°C. to constant weight. The loss is considered to be moisture.

TOTAL PHOSPHORIC ACID.

Gravimetric Method.

5 REAGENTS.

(*a*) *Molybdate reagent.* Dissolve 133 grm. ammonium molybdate in 800 c.c. water. Add an amount of ammonia which is just sufficient to dissolve the slight turbidity. Make up the solution to 1 litre. Dilute 487 c. c. of nitric acid (sp. gr. 1·416) to 1 litre. Just when required, mix equal volumes of ammonium molybdate and nitric acid solutions, always pouring the molybdate solution into the acid. This mixture will be hereafter referred to as molybdate reagent.

(*b*) *Ammonium nitrate solution.* Dissolve 200 grm. of commercial ammonium nitrate (phosphate-free) in water and dilute to 2 litres.

(*c*) *Magnesia mixture.* Dissolve 22 grm. of recently ignited calcined magnesia in dilute hydrochloric acid avoiding an excess of acid, add a little calcined magnesia in excess and boil for a few minutes to precipitate iron, aluminium and phosphoric acid. Filter, add 280 grm. of ammonium chloride, and 261 c. c. of ammonium hydrate (sp. gr. 0·90) and dilute to 2 litres. Instead of a solution of 22 grm. calcined magensia in dilute hydrochloric acid, 110 grm. of crystallized magnesium chloride, $MgCl_2 \cdot 6H_2O$, dissolved in water may be substituted.

(*d*) *Dilute ammonium hydrate for washing.* Dilute 100 c.c. of ammonium hydrate of 0·90 sp. gr. to 1 litre.

6 PREPARATION OF SOLUTION.

Treat 2 grm. of the sample by one of the methods given below. In the case of (*d*), 2·5 grm. may be used. Cool the solution, dilute to 200 c. c., or to 250 c. c. if a 2·5 grm. sample was used. Mix and pour on a dry filter.

(*a*) Ignite and dissolve in hydrochloric acid.

(*b*) Evaporate with 5 c. c. of magnesium nitrate, ignite and dissolve in hydrochloric acid.

Magnesium nitrate solution. Dissolve 320 grm. of calcined magnesia in nitric acid avoiding an excess of the latter, then add a little calcined magnesia in excess, boil, filter from the excess of magnesia, precipitated ferric oxide, etc., and dilute to 2 litres.

(*c*) Boil with 20-30 c. c. of strong sulphuric acid in a Kjeldahl flask adding 2-4 grm. of sodium or potassium nitrate at the beginning of the digestion and a small quantity after the solution has become nearly colourless, or adding the nitrate in small portions from time to time. After the solution is colourless, add 150 c. c. of water and boil for a few minutes.

(*d*) Digest in a Kjeldahl flask with strong sulphuric acid and such other reagents as are used in either the plain or modified Kjeldahl or Gunning method for estimating nitrogen. Do not add potassium permanganate but, after the solution has become colourless, add about 100 c. c. of water and boil for a few minutes.

(*e*) Dissolve in 30 c. c. of concentrated nitric acid and a small quantity of hydrochloric acid and boil until organic matter is destroyed.

(*f*) Add 30 c. c. of concentrated hydrochloric acid, heat and add cautiously, in small quantities at a time, about 0·5 grm. of

finely pulverized potassium chlorate to destroy organic matter.

(*g*) Dissolve in 15-30 c. c. of strong hydrochloric acid and 3-10 c. c. nitric acid. This method is recommended for fertilizers containing much iron or aluminium phosphate.

7 DETERMINATION.

In case hydrochloric or sulphuric acid has been used as a solvent, add about 15 grm. of dry ammonium nitrate or a solution containing that amount. To the hot solution add 70 c. c. of the molybdate reagent for every decigram of phosphoric acid (P_2O_5) present. Digest at about 65°C. for an hour, and determine if the phosphoric acid has been completely precipitated, by the addition of more molybdate reagent to the clear supernatant liquid. Filter and wash with ammonium nitrate solution. Dissolve the precipitate on the filter with ammonium hydroxide and hot water and wash into a beaker to a bulk of not more than 100 c. c. Nearly neutralize with hydrochloric acid, cool, and from a burette add slowly (about 1 drop per second) stirring vigorously, 15 c. c. of magnesia mixture for each decigram of phosphoric acid (P_2O_5) present. After 15 minutes add 12 c. c. of ammonium hydroxide (sp. gr. 0·90). Let stand till the supernatant liquid is clear (2 hours are usually enough), filter and wash with the dilute ammonium hydroxide until the washings are practically free from chlorides. Dry, ignite to whiteness or to a greyish white, weigh and calculate to phosphoric acid (P_2O_5).

8 *Volumetric method.*

A series of tests has been carried out at Pusa, on the strength of which the Committee recommend the following procedure.

9 REAGENTS.

(*a*) *Molybdate solution.* Shake 200 grm. of powdered ammonium molybdate with about 800 c. c. water in a stoppered cylinder. The salt dissolves at first but the solution soon turns milky. Then cautiously add a solution of ammonia (sp. gr. 0·91) to the molybdate solution till this turns clear. The solution should smell only very faintly of ammonia. Make up volume to 1 litre with distilled water. 1 c. c. of this solution is equivalent to 0·005 grm. phosphoric acid (P_2O_5).

The precipitating molybdate acid mixture is prepared immediately before use, by pouring a quantity of the above ammonium molybdate solution into an equal volume of strong nitric acid (sp. gr. 1·416).

(*b*) *Standard caustic soda solution.* Use N/7 or N/10 solution.

(*c*) *Standard nitric acid solution.* Use N/7 or N/10 solution.

(*d*) *Potassium nitrate solution.* Prepare a solution containing 3 grm. of potassium nitrate in 100 c. c. of water.

(*e*) *Phenol-phthalein solution.* Dissolve 1 grm. phenol-phthalein in 100 c. c. of 90 per cent. alcohol.

(*f*) *Blue litmus paper.*

10 PREPARATION OF SOLUTION.

Proceed as directed under **6**.

11 DETERMINATION.

Evaporate an aliquot part of the solution, containing from 5 to 40 mg. phosphoric acid (P_2O_5), down to dryness on the waterbath. Add a few drops of hydrochloric acid and a little water and again evaporate the solution to dryness. Treat the residue with dilute nitric acid and filter. Collect the filtrate and washings in a 100 c. c. beaker, make up to about 50 c. c. and pour 20 c. c. of the freshly prepared molybdic acid mixture into solution. Vigorously stir the contents of the beaker with a glass rod, taking care not to touch the sides. Keep the beaker in a warm place, where the temperature is maintained at about 35°C. Stir the contents at least 4 times, at intervals of about half an hour. At the end of 24 hours, filter off the supernatant liquid through paper contained in a small porcelain Gooch crucible and wash the precipitate 3 times by decantation with dilute nitric acid, using 3 c. c. each time, and passing the decanted liquid through the crucible. Continue the washing with the potassium nitrate solution, till the contents of the beaker are free of acid (as tested by litmus paper). Carefully wash the filter paper and the crucible with the nitrate solution till they are also free of nitric acid. Transfer the paper and the precipitate adhering to the crucible to the beaker with the help of a little distilled water. Add a measured volume of standard caustic soda solution till the phospho-molybdate precipitate is dissolved, and titrate back the excess of alkali with standard nitric acid, using phenol-phthalein as indicator.

Each cubic centimeter of N/7 alkali used =0·000441 grm. phosphoric acid (P_2O_5).

12 WATER-SOLUBLE PHOSPHORIC ACID.

Place 2 grm. of the sample on a 9 cm. filter. Wash with successive small portions of water, allowing each portion to pass through before adding more, until the filtrate measures nearly 250 c. c. If the filtrate is slightly turbid, clear with a little nitric acid. Make up to 250 c. c. Mix

thoroughly and determine the phosphoric acid (P_2O_5) in an aliquot portion of the solution by one of the methods described at 7 and 11 above.

CITRATE-INSOLUBLE PHOSPHORIC ACID.

13 PREPARATION OF AMMONIUM CITRATE SOLUTION.

Dissolve 370 grm. of citric acid in 1,500 c.c. of water and nearly neutralize with ammonia. Cool. Add ammonia until exactly neutral to corallin (saturated alcoholic solution). Dilute sufficiently to make the specific gravity 1·09 at 20°C.

14 DETERMINATION.

Heat 100 c. c. of ammonium citrate solution in a loosely stoppered flask to 65°C. by placing it in a warm water bath. When the solution has attained this temperature, introduce 2 grm. of sample which must be free of acid. Close the flask, shake and replace in the water-bath. Maintain at 65°C. mixing the contents vigorously every 5 minutes. At the expiration of exactly 30 minutes, remove the flask from the bath, filter the contents rapidly through a quick-acting filter, and wash repeatedly with water at 65°C. allowing the filter to drain completely each time. Stop the washing when about 350 c. c. of filtrate have passed through. Transfer the filter and its contents to a crucible ignite and digest with 10-15 c. c. strong hydrochloric acid until the phosphate is dissolved. If there is fear of loss of phosphoric acid during ignition, treat the filter paper and its contents with 30-35 c. c. strong nitric acid and 5-10 c. c. hydrochloric acid and boil until all the phosphate is dissolved. Dilute, filter, make up to 250 c. c. and determine the phosphoric acid by one of the methods described previously at 7 and 11.

If the sample is acidulated (*e.g.* superphosphate) employ the residue obtained from the determination of the water-soluble phosphoric acid. In this instance, drop the filter and residue from this treatment into the citrate solution, close the flask and shake until the filter is reduced to pulp. Then proceed as above.

15 CITRATE-SOLUBLE PHOSPHORIC ACID.

The citrate-soluble phosphoric acid is determined by difference. Subtract the sum of water-soluble and citrate-insoluble from the total phosphoric acid to get the citrate-soluble phosphoric acid.

ORGANIC AND AMMONIACAL NITROGEN ONLY.

Kjeldahl Method.

16 REAGENTS.

(*a*) *Standard sulphuric acid.* Dilute pure sulphuric acid with about half its volume of water, cool and determine its specific gravity. From the specific gravity the percentage of sulphuric acid (H_2SO) is calculated by Marshall's formula.

$P=S\ (85{\cdot}87+0{\cdot}05\ T-0{\cdot}0004\ t^2)-69{\cdot}82$ where P=percentage of H_2SO_4 by weight, S=specific gravity at T°C. compared with water at t°C. The formula holds good between 0°C. and 40°C. for acids containing 62—82 per cent. H_2SO_4. Calculate the weight of acid required to make up a standard solution from the formula $W=n\ A\times\frac{100}{P}$, where W=the weight of acid to be used, A=the number of grams of H_2SO_4 per litre in the required standard solution, and n=the number of litres of solution required. Weigh out W grams of acid and make up to n litres. N/7 acid is convenient for most fertilizers. Where small amounts of nitrogen have to be determined, N/10 acid may be used instead.

The strength of this acid is checked as follows :—

(1) Precipitate 25 c.c. of acid with barium chloride solution and weigh the barium sulphate formed.

(2) Weigh out accurately about 0·4 grm. of pure recrystallized ammonium chloride into a flask, add a slight excess of sodium or potassium hydrate and distill off the ammonia into 25 c.c. of the new standard acid. Titrate back the excess of acid by means of standard alkali and calculate the strength of the acid by noting the amount which is neutralized by the known weight of ammonia, evolved from the ammonium chloride taken.

(*b*) *Standard alkali.* Prepare a solution of caustic soda exactly equal in strength to the standard acid.

(*c*) *Sulphuric acid.* Use acid of specific gravity 1·84, free from nitrogen.

(*d*) *Potassium sulphate.* Pure, free from nitrogen.

(*e*) *Copper sulphate.* Crystallized.

(*f*) *Mercuric oxide.* Pure, free from nitrate.

(*g*) *Sodium sulphide.* Take a 30 per cent. solution of caustic soda and pass sulphuretted hydrogen until it is saturated.

(*h*) *Sodium hydroxide.* A solution of specific gravity 1·263 at 30°C, *i.e.*, approximately of 30 per cent. strength.

(*i*) *Methyl red solution.* Dissolve 1 grm. of methyl red in 100 c.c. of 95 per cent. alcohol.

(*j*) *Methyl orange solution.* Dissolve 0·5 grm. in 500 c.c. of distilled water.

17 DETERMINATION.

(*a*) *Pusa Method.* Place 0·7—3·5 grm. of substance, according to the nitrogen content of material to be analysed, in a Kjeldahl flask and add 1 grm. of copper sulphate and a suitable quantity (20—30 c.c.) of sulphuric acid. Apply heat, taking care that the preliminary heating is not forced. After the organic matter has gone into solution and the liberated water has been expelled, cool somewhat and add 10 grm. of anhydrous potassium sulphate, a little at a time. Heat the mixture until pure green in colour. Continue the boiling for 15 minutes after the liquid has become clear. Transfer the acid liquid to a 1·5 litres flask, together with the washings and make up the volume to about 500 c.c. Add the necessary quantity of soda solution, taking care to pour it down the side of the flask so as to avoid admixture of the two liquids at this stage. The alkali sinks to the bottom and the surface remains acid. When all connections have been made, mix the liquids and, by admitting steam, carry out the distillation in a fairly rapid manner. Collect 300 c.c. of the distillate in a measured excess of standard acid to which methyl red or methyl orange has been added to serve as indicator. Titrate the excess of acid with standard alkali.

Previous to use, the reagents should be tested by a blank experiment with sugar or filter paper.

(*b*) *Dyer's method.* This method has been found satisfactory for the determination of organic nitrogen in refractory substances. Place 0·7—3·5 grm. of substance, according to its nitrogen content, in a Kjeldahl flask. Add 0·7 grm. of mercuric oxide and then 25 c.c. of sulphuric acid and mix. Heat up gently. After the organic matter has gone into solution and the liberated water expelled, cool somewhat, add 10 grm. powdered potassium sulphate and proceed as directed under (*a*) above, except that when proceeding to distil off the ammonia, 2 c.c. of sodium sulphide solution are to be added to the soda solution.

TOTAL NITROGEN.

Kjeldahl Method modified to include the Nitrogen of Nitrates.

18 REAGENTS.

In addition to those specified under **16**, the following reagents are required.

Sulphuric acid containing salicylic acid. Add 1 gram salicylic acid to every 30 c.c. of nitrogen-free strong sulphuric acid.

Sodium thiosulphate. Recrystallized.

19 DETERMINATION.

Place 0·7—3·5 grm. according to the nitrogen content of the material, in a Kjeldahl flask. Add 30—40 c.c. of the sulphuric acid containing salicylic acid, shake until thoroughly mixed, and allow to stand for at least 30 minutes. Add 5 grm. of crystallized sodium thiosulphate, mix, place in the heating stand and heat over a low flame until all danger of frothing has passed. Increase the flame until the acid boils briskly and continue the boiling until white fumes no longer escape from the flask. This requires about 10 minutes. Add approximately 1 grm. of copper sulphate and continue the boiling until the organic matter has gone into solution. Next add 10 grm. of potassium sulphate, a little at a time. Boil and continue the boiling for at least 15 minutes after the liquid in the flask is quite clear. The distillation and titration are then carried out as usual.

20 AMMONIACAL NITROGEN.

Place 0·7—3·5 grm. according to the nitrogen content of the material, along with 200 c.c. of water and 5 grm. or more of magnesium oxide (free from carbonate), in a flask connected to a condenser. Distil 100 c.c. of the liquid into a measured quantity of standard acid and titrate back with standard alkali.

21 NITRIC AND AMMONIACAL NITROGEN.

Place 1 grm. of the sample, containing not more nitrate than the equivalent of 0·25 grm. potassium nitrate, in a 500 c.c. Kjeldahl flask. Add 30 c.c. of water and then introduce 5 grm. of reduced iron in small portions at a time. Mix to ensure complete solution of the soluble nitrate and ammonium salts. Add 10 c.c. of a mixture of equal volumes of sulphuric acid and water. Place a long-stemmed funnel in the neck of the flask to prevent mechanical loss and gently mix. Allow to stand until the violence of the reaction is over. Next apply heat slowly and boil for 5 minutes. Cool and transfer the contents into a distillation flask of 1·5 litres capacity. Add 30 c.c. of a 30 per cent. solution of caustic soda and distil with steam through a condenser into standard acid. The titration figures give the total nitrogen present as ammonium salts and nitrates.

POTASH.

Platinum Method.

22 REAGENTS.

(*a*) *Ammonium chloride solution.* Dissolve 100 grm. of ammonium chloride in 500 c.c. of water, add from 5–10 grm. of pulverized potassium, platinic chloride and shake at intervals for 6—8 hours. Allow the mixture to settle overnight and filter just before use. The residue may be used for the preparation of a fresh supply.

(*b*) *Platinum solution.* Use a solution containing the equivalent of 1 grm. of metallic platinum (2·1 grm. H_2PtCl_6) in every 10 c.c.

(*c*) *73·59 per cent. alcohol.* Sp. gr. 0·8639 at 60°F./60°F., or 0·8593 at 20°C./1°C. This strength is equal to 80 per cent. by volume.

23 PREPARATION OF SOLUTION.

(*a*) *Mixed fertilizers; mixtures containing potash salt, superphosphate, wood ashes, etc.* Weigh out 5 grm. of the sample on a 12·5 cm, filter paper and wash with successive small amounts of boiling water until the filtrate amounts to about 400 c.c. Add to the hot solution a slight excess of ammonium hydroxide and then sufficient ammonium oxalate to precipitate all the lime present. Cool, dilute to 500 c.c., mix and pass through a dry filter before analysis.

(*b*) *Soluble potash salts; sulphate or muriate of potash kainite, sulphate of potash and magnesia.* Weigh out 5 grm. of the sample, transfer to a 12·5 cm. filter paper and wash with successive small quantities of boiling water until the volume is about 400 c.c. Cool, and make up to 500 c.c.

(*c*) *Organic substances; oilcakes, tobacco stems, etc.* To determine the total potash in organic fertilizers, saturate 10 grm. with strong sulphuric acid, and ignite in a muffle at a low red heat to destroy organic matter. Cool. Add a little strong hydrochloric acid, warm slightly to loosen the mass from the dish, dissolve in water and wash with hot water into a 500 c.c. graduated flask until the volume is about 400 c.c. Add to the hot solution ammonia and ammonium oxalate as in(*a*), cool and finally dilute to 500 c.c. Mix and pass through a dry filter before analysis.

24 DETERMINATION.

(*a*) *Mixed fertilizers.* Evaporate 50 c.c. of the solution, made according to (*a*) above, corresponding to 0·5 grm. of the sample

nearly to dryness, add 1 c.c. of dilute sulphuric acid (1 to 1), evaporate to dryness and ignite to whiteness. As all the potash is in the form of sulphate no loss by volatilization need be apprehended and a full red heat must be maintained until the residue is perfectly white. Dissolve the residue in hot water using at least 20 c.c. for each decigram of potash (K_2O). Add a few drops of hydrochloric acid and then platinum solution in excess. Evaporate on water-bath to a thick paste and treat the residue with 80 per cent. alcohol, avoiding exposure to ammonia. Wash the precipitate thoroughly with 80 per cent. alcohol both by decantation and on the filter, continuing the washing after the filtrate is colourless. Next wash with 10 c.c. of the ammonium chloride solution, to remove impurities from the precipitate and repeat 5 or 6 times. Wash again thoroughly with 80 per cent. alcohol and dry the precipitate for 30 minutes at 100°C. The precipitate should be perfectly soluble in water.

(*b*) *Water-soluble potash in wood ashes, etc.* Prepare the solution as directed in **23** (*a*), and determine the potash as in (*a*) above, paying special attention to the last sentence of (*a*).

(*c*) *Muriate of potash.* Take 50 c.c. of the solution prepared as directed under **23** (*b*), acidify with a few drops of hydrochloric acid, add 10 c.c. of the platinum solution and evaporate to thick paste. Then proceed as under (*a*) above.

(*d*) *Sulphate of potash, sulphate of potash and magnesia and kainite.* Take 50 c.c. of solution prepared as under **23** (*b*). Acidify with a few drops of hydrochloric acid and add 15 c.c. of platinum solution. Evaporate the solution and proceed as under (*a*) above, except that 25 c.c. portions of ammonium chloride solution should be used.

(*e*) *Organic compounds; oil cakes, tobacco stems, etc.* Prepare the solution as directed in **23** (*c*) and determine the potash as in (*a*) above, paying special attention to the last sentence of (*a*).

For the conversion of potassium platinic chloride (K_2PtCl_6) to potassium chloride (KCl) use the factor 0·3067; to potassium sulphate (K_2SO), 0·3585; to potassium oxide (K_2O), 0·1938. (Calculations based on atomic weight of platinum=195·2).

Perchlorate method.

25 REAGENTS.

(*a*) *Perchloric acid.* A solution of 20 per cent. strength (sp. gr. 1·12) is required. Care should be taken to ascertain that the perchloric acid

used is free from chloric acid [Page, "On the Perchlorate Method for the Estimation of Potassium in Soils, Fertilizers, etc.," *Jour. Agri. Sci.*, 14, (1924), 133]. Some samples of perchloric acid on the market contain traces of potash salts and also traces of sulphuric acid or sulphates. In view of this fact, it is necessary to make blank determinations, which should be carried out as follows :—

(1) *Potash salts.* Evaporate 10 c.c. of the 20 per cent. perchloric acid nearly to dryness. Take up with 20 c.c. of 95 per cent. alcohol and after letting it stand for one hour filter the insoluble matter, if any, on a weighed Gooch crucible. Wash with 5 c.c. of 98 per cent. alcohol to remove traces of perchloric acid and then with 100 c.c. of alcohol saturated with potassium perchlorate. Dry to constant weight. The amount of potassium perchlorate, if any, found in this blank determination with 10 c.c. of perchloric acid used, should be deducted from the weight of the perchlorate found in the analysis. "Pure" perchloric acid has been known to contain 0·0010—0·0015 grm. potassium perchlorate per 10 c.c., and sometimes even larger amounts have been noted.

(2) *Sulphuric acid or sulphates.* If the perchloric acid contains a trace of sulphuric acid, a turbidity due to barium sulphate appears on adding 5 c.c. of perchloric acid to the solution filtered from the baryta treatment and evaporating. Traces of sulphuric acid, formed by the combustion of coal gas from the burners used to heat the sand-bath, may also produce a slight precipitate of barium sulphate during the evaporation. The error from these causes is most simply obviated as follows. After drying and weighing the perchlorate in the Gooch crucible, dissolve the precipitate by washing with 300 c.c. of boiling water. Weigh the Gooch crucible after drying it at 120° C., and compare its weight with that before collecting the precipitate. If there is any increase in weight, due to insoluble barium sulphate, the second weight (after the washing with boiling water) should be used to calculate the weight of the perchlorate. This procedure does not waste any time in practical working, as the perchlorate in a series of analyses is weighed in the same Gooch crucible, each lot of perchlorate after weighing being washed away with boiling water.

(*b*) *Baryta solution.* Prepare a solution of 3 per cent. strength.

(*c*) 95 *per cent. alcohol.*

(*d*) 98 *per cent. alcohol.*

(*c*) *Alcohol saturated with potassium perchlorate.* Put ordinary 95 per cent. alcohol into a Winchester quart, along with about 2—3 grm. of finely powdered pure potassium perchlorate, obtained from previous analyses. Shake the mixture several times during 48 hours so as to fully saturate it. The solution is then ready for use. Every time the alcohol is wanted for washing purposes, thoroughly shake the contents of the bottle and filter the alcohol into a wash bottle. This ensures the washing liquid being saturated at the actual temperature of working. The alcohol should be perfectly clear. The test for proper saturation is that when 100 c.c. of it is used to wash a precipitate of pure potassium perchlorate, the loss of weight is less than 0·0001 grm.

26 PREPARATION OF SOLUTION.

Proceed as directed under **23**.

27 DETERMINATION.

(*a*) *Sulphate of potash, kainite, sulphate of potash and magnesia, and mixed fertilizers.* Transfer 50 c.c. of the solution, prepared as directed under **23** (*b*) and representing 0·5 grm. of the original sample, to a 7 cm. porcelain dish, add 15 c.c. of barium hydroxide solution, warm, add ammonium carbonate solution to precipitate the excess of barium, filter and evaporate the filtrate to dryness on a steam bath. Ignite the residue over a Bunsen burner below a red heat for 15 minutes. Extract the residue with boiling water, breaking up the material with a small glass pestle made from a piece of ¼ inch glass rod, filter into an evaporating dish (preferably of Jena or Resistance glass) of about 175 c.c. capacity and wash with boiling water until the filtrate amounts to about 150 c.c. Add 5 c.c. of 20 per cent. perchloric acid (sp. gr. 1·12) and evaporate on steam or sand bath until it fumes strongly. Take up the residue with 10 c.c. of water, add a second 5 c.c. of perchloric acid and again evaporate the solution on the sand bath until all free hydrochloric acid is driven off and dense white fumes of perchloric acid appear. Cool, add 20 c.c. of 95 per cent. alcohol, stir, allow to stand half an hour, and decant the whole of the alcohol through a weighed filter paper, or preferably a Gooch crucible, draining as thoroughly as possible. Wash the precipitate on to the filter paper or Gooch crucible with the help of 5 c.c. of 98 per cent. alcohol in order to remove traces of perchloric acid. Thoroughly wash with about 125 c.c. of 95 per cent. alcohol previously saturated with pure potassium perchlorate at the temperature of working. Dry to constant weight at 120°C. and weigh. Wash again with another 50 c.c. of saturated alcohol and again weigh. If the loss

in weight exceeds 0·0005 grm., rewash the precipitate until constant in weight. After weighing, dissolve the precipitate from the crucible in hot water and again weigh for the next analysis. There should at this stage be no insoluble residue. If such is present, it is due to the occurrence of sulphates or sulphuric acid as an impurity in the perchloric acid used giving rise to barium sulphate [*see* **25** (*a*) (2) above]. In such cases the weight of the insoluble residue should be deducted to obtain the weight of true perchlorate.

NOTE.—Care must be taken not to evaporate the solutions to which baryta has been added, in the same hood as those in which the final solution containing perchloric acid are being evaporated. If ammonium salts are present in the former solutions, they give off ammonia which is taken up by the perchloric acid solutions, forming ammonium perchlorate, which behaves like potassium perchlorate.

For the conversion of potassium perchlorate ($KClO_4$) to potassium chloride (KCl) use the factor 0·5381; to potassium sulphate (K_2SO_4), 0·6289; to potassium oxide (K_2O), 0·3400.

(*b*) *Muriate of potash.* Proceed as directed under (*a*) above, but the treatment with baryta can be omitted.

(*c*) *Organic fertilizers.* Take 50 c.c. of the solution prepared as in **23** (*c*) and proceed as directed under (*a*) above.

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